

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization International Bureau



(43) International Publication Date
20 January 2005 (20.01.2005)

PCT

(10) International Publication Number
WO 2005/005673 A2

- | | | |
|--|--|--|
| (51) International Patent Classification ⁷ : | C22B 9/00 | (81) Designated States (<i>unless otherwise indicated, for every kind of national protection available</i>): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW. |
| (21) International Application Number: | PCT/EP2004/007389 | |
| (22) International Filing Date: | 6 July 2004 (06.07.2004) | |
| (25) Filing Language: | English | |
| (26) Publication Language: | English | |
| (30) Priority Data: | 2003120999 | 8 July 2003 (08.07.2003) RU |
| (71) Applicant (<i>for all designated States except US</i>): | LINDE AKTIENGESELLSCHAFT [DE/DE]; Abraham-Lincoln-Str. 21, 65189 Wiesbaden (DE). | |
| (72) Inventor; and | | |
| (75) Inventor/Applicant (<i>for US only</i>): | VOROTYNTSEV, Vladimir, Michailovich [RU/RU]; 1-46, Pjatogorskaja str., Nizhny Novgorod, 603009 (RU). | |
| (74) Common Representative: | LINDE AKTIENGESELLSCHAFT; Abraham-Lincoln-Str.21, 65189 Wiesbaden (DE). | |
- Published:**
— without international search report and to be republished upon receipt of that report
- For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

(54) Title: METHOD FOR PREPARING HIGH-PURITY GERMANIUM HYDRIDE

A2 (57) **Abstract:** The invention relates to the preparation of germanium-containing materials and concerns the development of an electrochemical method for preparing high-purity germanium hydride, suitable for use as a source of germanium in microelectronics technologies. Germanium hydride is prepared by electrolysis of an aqueous-alkaline solution, containing germanium dioxide in a concentration of from not less than 40 g/l to the solubility limit, at a nickel cathode in a diaphragm cell at a current density of 1.0-1.5 A/cm² and a temperature no higher than 65°C, first passing an electrical current through the aqueous-alkaline solution for the time needed to achieve the minimum possible content of contaminants limiting for germanium hydride. The electrolysis is performed with cross-mixing of electrolyte streams, feeding a stream of electrolyte from the cathode chamber, after removal of germanium hydride and hydrogen, into the anode chamber, and a stream of electrolyte from the anode chamber, after removal of oxygen, into the cathode chamber. The germanium hydride obtained after synthesis is isolated from the mixture with hydrogen. For more thorough purification, the isolated germanium hydride is purified by the membrane method. The technical result is preparation of germanium hydride in which the total content of the contaminants SiH₄, AsH₃, PH₃, H₂S, CH₄, Fe, Ni, Al, Ca, Mg, etc. is not more than 1·10⁻⁶% - 1·10⁻⁷%, which is acceptable for comparatively wide fields of practical application. The use of the membrane method ensures removal from the germanium hydride of suspended particles with a size of 0.05 µm to a level of less than 5.5·10⁻³ particles/mole, making it suitable in such fields as, for example, optics and laser engineering. The productivity of the method is 40-50 g/hour. 2 main claims, 8 dependent claims, 1 example.

WO 2005/005673 A2